PROCESS FOR MAKING FINELY DIVIDED, DENSE PACKING, SPHERICAL SHAPED SILVER PARTICLES

CROSS REFERENCE TO RELATED APPLICATION

This application is a continuation-in-part of U.S. patent application Ser. No. 08/089,031, filed Jul. 13, 1993, now abandoned.

FIELD OF THE INVENTION

The invention is directed to an improved process for making finely divided silver particles. In particular, the invention is directed to a process for making silver powders that are finely divided, dense packing spheres.

BACKGROUND OF THE INVENTION

Silver powder is used in the electronics industry for the manufacture of conductor thick film pastes. The thick film pastes are screen printed onto substrates forming conductive circuit patterns. These circuits are then dried and fired to volatilize the liquid organic vehicle and sinter the silver particles.

Printed circuit technology is requiring denser and more precise electronic circuits. To meet these requirements, the conductive lines have become more narrow in width with smaller distances between lines. The silver powders necessary to form dense, closely packed, narrow lines must be as close as possible to monosized, dense packing spheres.

Many methods currently used to manufacture metal powders can be applied to the production of silver powders. For example, thermal decomposition processes, electrochemical processes, physical processes such as atomization or milling, and chemical reduction methods can be used. Thermal decomposition processes tend to produce powders that are spongy, agglomerated, and very porous whereas electrochemical processes produce powders that are crystalline in shape and very large. Physical processes are generally used to make flaked materials or very large spherical particles. Chemical precipitation processes produce silver powders with a range of sizes and shapes.

Silver powders used in electronic applications are generally manufactured using chemical precipitation processes. Silver powder is produced by chemical reduction in which an aqueous solution of a soluble salt of silver is reacted with an appropriate reducing agent under conditions such that ionic silver is reduced and silver powder is precipitated. Inorganic reducing agents including hydrazine, sulfite salts and formate salts produce powders which are very coarse in size, are irregularly shaped and have a large particle size distribution due to aggregation.

Organic reducing agents such as alcohols, sugars or aldehydes are used to reduce silver nitrate in the presence of a base such as alkali hydroxides or carbonates. See *Silver—Economics, Metallurgy and Use,* A. Butts, ed. 1975, Krieger Publishing Co., N.Y., p. 441. The reduction reaction is very fast, hard to control and produces a powder contaminated with residual alkali ions. Although small in size (e.g., <1 micron), these powders tend to have an irregular shape with a wide distribution of particle sizes that do not pack well. These types of silver powders exhibit difficult to control sintering and inadequate line resolution in thick film conductor circuits.

PRIOR ART

U.S. Pat. No. 4,078,918 1978 Perman

A recovery process for reclaiming precious metals from industrial process residues, such as silver chloride resulting from salt analysis of meats in a packing plant, or alternative, from industrial waste photographic papers or the like. The process comprises pretreating the material with an oxidizing agent capable of substantially completely oxidizing organic contaminants, reacting the material with ammonium hydroxide to form a soluble ammonia complex, and reacting the ammonia complex with ascorbic acid or a salt form of ascorbic acid to provide precious metal in elemental form. The preferred process is for reclaiming silver.

European Patent Application 0 073 108 1981 Perrin

A process for the recovery of metals from solutions containing them, particularly for recovering gold, silver, platinum or other precious metals in a pure from, comprises the use of a reduction reaction using as reducing agent a polyhydroxyl compound. Suitable polyhydroxyl compounds are sugars, particularly those having a lactone structure, for example L-ascorbic, D-isoascorbic acid and salts thereof.

U.S. Pat. No. 4,863,510 1989 Tamemasa et al.

Fine particles of a metal such as copper and silver can be obtained by reducing the corresponding metal ammonium complex salt solution with one or more reducing agents selected from the group consisting of L-ascorbic acid, L-ascorbate, D-erythorbic acid and D-erythorbate.

SUMMARY OF THE INVENTION

This invention is directed to a method for the preparation of finely divided, dense packing, spherical shaped silver particles comprising the sequential steps of

(1) reacting an aqueous mixture of a silver salt with an alkanolamine to form a homogeneous aqueous solution of a dissolved silver alkanolamine complex;

(2) preparing an aqueous solution of a reducing agent and, optionally an alkanolamine; and

(3) mixing together the silver alkanolamine complex solution and the reducing agent solution at a buffered pH and a temperature between 10° C. to 100° to form finely divided, dense packing, spherical silver particles.

DETAILED DESCRIPTION OF THE INVENTION

The process of the invention is a reductive process in which finely divided, dense packing, spherical silver particles are precipitated by adding together an aqueous solution of a silver alkanolamine complex and an aqueous solution containing the mixture of a reducing agent and an alkanolamine. Finely divided is defined as non-agglomerated with a narrow particle size distribution, dense packing is indicated by large tap density, and spherical shape is determined by scanning electron microscopy.

The silver alkanolamine complex aqueous solution is prepared by first adding a water-soluble silver salt to deionized water to form an aqueous silver mixture. Any water-soluble silver salt can be used in the process of the invention such as silver nitrate, silver phosphate, and silver sulfate. Addition of an alkanolamine to the aqueous silver mixture produces an aqueous solution of